

PATENT SPECIFICATION

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(54) PROCESS FOR THE MANUFACTURE OF
 POLYETHYLENE TEREPHTHALATE GRANULES

(71) We, FARBWERKE HOECHST AK-
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 under German Law, of 6230 Frankfurt (M)-
 Hoechst, Germany, do hereby declare the in-
 vention, for which we pray that a patent may
 be granted to us, and the method by which
 it is to be performed, to be particularly de-
 scribed in and by the following statement:—

The present invention relates to a pro-
 cess for the manufacture of polyethylene
 terephthalate granules which, after exposure
 to a thermal treatment in the solid phase, may
 be worked up by injection-moulding.

Polyethylene terephthalate acquires out-
 standing mechanical properties after exposure
 to a thermal treatment in the solid phase at
 temperatures above 200°C owing to the re-
 sulting condensation to higher molecular
 weights and this plastics material in the highly
 condensed form can be worked up by in-
 jection - moulding. However, when being
 worked up in injection machines, the unpro-
 cessed plastics material has to meet certain
 standards of shape. It must, for example, be
 free flowing, it must neither stick nor have
 a tendency to bridging since otherwise the
 uniform feeding of the plastics material into
 the injection machine is not assured. The
 conventional granulating processes applied to
 polyethylene glycol terephthalate, for example
 continuous granulation, usually yield granular
 products, so-called chips, which are not suit-
 able for being worked up in injection
 machines. Polyesters having a specific vis-
 cosity of approximately 800, which are used
 as basic materials for the thermal condensa-
 tion in the solid phase, can be extruded by
 means of the conventional extruders with only
 great difficulty, for example into strand-shaped
 granules. A polyester having a specific vis-
 cosity of, for example, 1400 or 1500, which
 has undergone a thermal treatment, has a
 better behaviour in this respect. It can be
 worked up without difficulty into strand-
 shaped granules so that it is easy, after a

thermal treatment has been carried out, to
 obtain the desired granular shape. However,
 this method requires the additional expensive
 process steps of re-melting and extruding
 and results in moreover, a partial degrada-
 tion of the polyethylene terephthalate where-
 by the result of the thermal treatment is par-
 tially rendered ineffective.

The present invention provides a process
 for the manufacture of polyethylene tereph-
 thalate granules suitable for injection mould-
 ing, which process comprises transforming a
 polyethylene terephthalate melt into granules
 by means of an underwater granulator, and
 subjecting these granules to thermal treat-
 ment in the solid phase at a temperature
 greater than 200°C.

The basic material to be granulated is
 obtained by a conventional melt condensation
 and generally has a specific viscosity within
 the range of from 700 to 1000, preferably
 of from 800 to 850. The polyester melt, which
 preferably has a temperature of approxi-
 mately 280°C., is conducted to an underwater
 granulator being designed, for example, such
 that the hot strand of plastics material ex-
 truded from the die is seized by a water jet
 issuing from a tubular die, cooled, and passed
 through an underwater cooling zone to a
 cutting device. The length and the cross-
 section of the cylindrical granules formed can
 be controlled by the speed of the cutting de-
 vice and by the draw-off speed of the water.
 The wet granular product is passed over a
 draining screen, and then dried.

The granules obtained by the above granu-
 lation process each have, for example, the
 shape of a cylinder or a slightly flattened
 cylinder, with a length of from 3 to 4 mm
 and a diameter of from 2.5 to 3 mm. The
 special advantage of the above granulation
 process is that a granular product of a very
 uniform shape is obtained without dust and
 cuttings, and this product is extremely suit-
 able for a subsequent thermal condensation
 in the solid phase at a temperature above

200°C. to form high molecular weight polyesters having a molecular weight of about 15000 to 25000 and a specific viscosity of about 1400 to 1500. The polyesters so obtained are particularly suitable for being worked up by injection moulding.

EXAMPLE

From an agitator vessel, in which 1.6 tons of polyethylene terephthalate melt had been highly condensed *in vacuo* to a specific viscosity of approximately 800, the melt, which had a temperature of 280°C., was forced out by means of a nitrogen pressure of 10 atmospheres gauge during the course of approximately 45 minutes, through an underwater granulator installed at the bottom of the vessel, said granulator consisting of 36 die holes and the same number of water jets. Each die hole had a circular opening of a diameter of 6 mm; the total throughput of water for taking away and cooling the strand amounted to 150 cu.m./h. The issuing strands, after having passed through a cooling zone of approximately 4 m length, were cut up by a cutting device whose speed was adjusted such that a granular product of a length of 3 mm was obtained. The diameter of the cylindrical granules was 2.5 to 3 mm. On issuing from the cutting device, the granules were passed over a screen to separate the solid material from the water, the latter being recirculated *via* a condenser. The wet granular product was, after drying, subjected to a thermal post-condensation.

WHAT WE CLAIM IS:—

1. A process for the manufacture of polyethylene terephthalate granules suitable for injection moulding, which process comprises transforming a polyethylene terephthalate melt into granules by means of an underwater granulator, and subjecting these granules to thermal treatment in the solid phase at a temperature greater than 200°C.

2. A process as claimed in claim 1, wherein the polyethylene terephthalate melt has a specific viscosity within the range of from 700 to 1000.

3. A process as claimed in claim 2, wherein the specific viscosity is from 800 to 850.

4. A process as claimed in any one of claims 1 to 3, wherein the polyethylene terephthalate melt has a temperature of approximately 280°C.

5. A process as claimed in any one of claims 1 to 4, wherein each of the granules (before thermal treatment) has the shape of a cylinder or a slightly flattened cylinder with a length of from 3 to 4 mm and a diameter of from 2.5 to 3 mm.

6. A process as claimed in claim 1, conducted substantially as herein described.

7. Polyethylene terephthalate granules whenever prepared and treated by a process as claimed in any one of claims 1 to 6.

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